

## PATENT SPECIFICATION

(11)

1 455 663

1 455 663

- (21) Application No. 13255/74 (22) Filed 26 March 1974  
 (31) Convention Application No. 2 315 114 (32) Filed 27 March 1973 in  
 (33) Germany (DT)  
 (44) Complete Specification published 17 Nov. 1976  
 (51) INT. CL.<sup>2</sup> B01F 3/10 5/00  
 (52) Index at acceptance  
 B1C 14 19A3 19F4B



(54) PROCESS AND APPARATUS FOR MIXING LIQUID  
 MATERIALS SHOWING GREAT DIFFERENCES IN VISCOSITY

(71) We, BASF AKTIENGESELLSCHAFT, a German Joint Stock Company of 6700 Ludwigshafen, Federal Republic of Germany, do hereby declare the invention, for which we pray that a Patent may be granted to us, and the method by which it is to be performed to be particularly described in and by the following Statement:—

The present invention relates to a process and apparatus for mixing liquid material showing viscosity differences of at least 2 tenth powers (in terms of poise with premixing effected in a side stream. In particular, this process is suitable for mixing low-viscosity materials such as lubricants, expanding agents, stabilizers and plasticizers with molten polymer.

Hitherto there have been three main ways of mixing low-viscosity substances with molten polymers. The first of these methods comprises adding the low-viscosity materials to the monomers and polymerizing them therewith immediately. On account of the influence on the polymerization conditions, evaporation losses and losses of said materials in subsequent operations (e.g. extraction), this method of mixing is only possible in special cases.

A widely used method, however, is to add the low-viscosity materials to the fully polymerized and molten polymer by the use of dynamic mixers. However, this method requires the use of relatively expensive machines such as screw machines and may lead to undesirable changes in the properties of the product or even to impairment of the product on account of the high shear stresses which occur in the polymer. Where the polymer throughput is high, it is necessary to divide the stream of molten polymer up into a number of streams flowing through separate dynamic mixers operating in parallel, due to the fact that these mixers have capacity limits imposed by their design. Mixing of low-viscosity materials with

high-viscosity substances by the use of static mixers, which may be enlarged to any desired extent, operate at low shear gradients and are thus less harmful to the product and which are superior to screw machines mainly in their lower price and running costs and also in their lower degree of trouble proneness, is highly unsatisfactory when the and also between the viscosities of the materials being mixed is more than 2 tenth powers in terms of poise.

Finally, it is possible to work the low-viscosity substances into the molten polymer after remelting a granular polymer. In this method, a polymer which may already have existed in the form of a melt must be remelted and this means that the properties of the product may be impaired and it is necessary to produce not only energy for mixing but also energy for remelting the polymer.

According to the invention, there is provided a process for mixing liquid materials with each other, which materials show a viscosity difference of at least 2 powers of ten (in terms of poise), by producing a premix in a branch stream of the high viscosity material, wherein at least a portion of the low-viscosity material is premixed with a portion of the high-viscosity material in the branch stream at a shear rate of at least 0.1 sec<sup>-1</sup> to form a premix having a content of the low-viscosity material of not more than 60% by weight of the premix, the premix is united with the main stream formed by the remainder of the high-viscosity material and the total stream thus formed is divided into partial streams which are spatially rearranged and are intimately mixed at a shear rate of not more than 10<sup>4</sup> sec<sup>-1</sup>.

An advantageous embodiment of the process of the invention consists in adding a second high-viscosity material via a side stream to the low-viscosity/high viscosity stream premixing stage. This second high-

2

1 455 663

2

viscosity substance fed through a side stream may, if desired, alternatively be added to the low-viscosity material alone.

A portion of the low-viscosity material may be fed directly to the high-viscosity main stream, whilst the remainder of the low-viscosity substance is added to the branch stream of high-viscosity substance and/or the side stream formed by the second high-viscosity substance.

A preferred embodiment of the process of the invention consists in that a portion of the low-viscosity substance is added to the high-viscosity substance(s) before or during manufacture thereof, whilst the remainder of the low-viscosity substance is mixed with the high-viscosity substance(s). In addition, high-viscosity additives with or without solids contents may be added to the branch stream and/or side stream and/or main stream.

The high-viscosity branch stream and/or side stream and the low-viscosity total stream or partial stream bear ratios to each other such that solubility limits are not or are only slightly exceeded and such that homogeneous mixing of the branch stream and/or side stream and the low-viscosity total stream and/or partial stream is possible at a shear gradient of at least  $0.1 \text{ sec}^{-1}$  and that the differences in viscosity between the premix obtained in the present manner and the high-viscosity substance contained in the main stream is not more than 2 tenth powers in terms of poise.

The present invention also relates to apparatus for carrying out the process of the invention, which is characterized in that a pipeline branching from the main line is connected, if desired together with a second pipeline for a second high-viscosity material, via a metering unit to a line which contains a second metering unit for the low-viscosity material, which junction point is connected to a dynamic mixer by a pipe, a further line connecting this mixer to the main line, which is in turn connected to a static mixer and may contain one or more additional connections for further pipelines.

Compared with prior art methods of mixing low-viscosity additives with molten polymers, the present process has the following advantages.

(1) Since large amounts of low-viscosity material are metered to the high-viscosity branch stream and/or side stream, a medium viscosity is produced in the dynamic mixer, this being up to 2 tenth powers (in terms of poise) lower than the viscosity of the high-viscosity material in the main stream. This reduced viscosity causes a reduction in the energy requirements of the dynamic mixer, and the premix is subjected to less mechanical and thermal stresses on account of the lower

degree of shear.

(2) The fact that the premix to be worked into the high-viscosity main stream has a viscosity which differs from that of the main stream by not more than 2 tenth powers (in terms of poise) makes it possible to achieve homogeneous mixing using a static mixer with all its advantages (low degree of trouble proneness due to the absence of moving parts, enlargability to any desired extent, extremely low price and running costs).

(3) In the case of products which are thermally sensitive, the process temperature may be reduced. The relatively low medium viscosity of the premix will thus be caused to rise in the dynamic mixer. Consequently, the temperature of the main stream may be reduced to an extent such that the viscosity difference remains below the limit of 2 tenth powers (in terms of poise). Low working temperatures mean reduced heat-energy requirements and, above all, a lessening of the risk of thermal deterioration of the product.

(4) On account of the lower viscosity of the premix in the dynamic mixer, the mixing units used in this mixing stage may be simpler and thus cheaper than twin-screw extruders and are for example single-screw extruders and toothed disc mixers. Depending on the percentage of low-viscosity material in the final product, only a relatively small partial stream is passed through the dynamic mixer, which may thus be of small size, i.e. a size just large enough to cope with the small throughputs. The main stream, which has the greatest volume, and the premix are however mixed in a static mixer which may be enlarged to any desired extent, such enlargement being a cheap matter. The combination of static and dynamic mixers used in the process of the invention gives the highly attractive advantage of low capital outlay and low running costs, particularly when compared with methods of mixing in low-viscosity additives in screw extruders, which allow only limited throughputs, due to their design, and may necessitate a number of parallel mixers.

An embodiment of an apparatus for carrying out the process of the invention is illustrated below with reference to the accompanying drawing.

At a point 2 on a pipeline 1 carrying a first high-viscosity material there is connected a pipeline 4 to carry a branch stream of said high-viscosity material, and said pipeline 4 may or may not be connected to a pipeline 3 carrying a side stream of a second high-viscosity material, before said pipeline 4 reaches a metering unit 5, e.g. a gear

pump. The line 4 leaving the metering unit 5 is connected to a pipeline 7 carrying all or some of the low-viscosity material passing through a further metering unit 8, e.g. a piston pump. This junction 6 is also connected to pipelines 14, 13, which may, if desired, carry liquid high-viscosity materials with or without solids contents. All of the pipelines 4, 7 and 13 meeting at junction 6 are connected via a pipeline 11 to a dynamic mixer 12, e.g. a toothed disc mixer, in which a homogeneous mixture of the streams of material is produced. The premix thus obtained passes from the dynamic mixer 12 through a pipeline 16 back to the main line 1 at junction 17, from which point the main line 1 carries premix and the remainder of the first high-viscosity material. At junction 18, the main line is connected to a pipeline 14, 15, through which further liquid high-viscosity materials with or without solids contents may be passed, and at junction 19 the main line 1 is connected to a pipeline 9 through which the remainder (if any) of the low-viscosity material is passed. The main line 1 is then connected to a static mixer 20 in which all of the materials passing through main line 1 are homogeneously mixed. A portion of the low-viscosity material may be fed to the static mixer 20 direct via a line 9, 10. The discharge line 21 connected to the static mixer 20 contains a homogeneous mixture of all of the high-viscosity and low-viscosity materials.

#### EXAMPLE 1

##### *Mixing of low-viscosity material (I) with nylon 6 (II)*

A stream of material I flowing at a rate of 150 g/hr and having a viscosity of about 10 poise (measured at 140°C) is added to a branch stream of polymer II flowing at a rate of 2.5 kg/hr and having a viscosity of about 2,000 poise (measured at 260°C), in a dynamic mixer at a temperature of about 260°C. The viscosity of the resulting premix is almost 1 tenth power less than that of the main stream of polymer II. The premix consisting of polymer branch stream and material I is then mixed at 251°C with the polymer main stream flowing at a rate of 2.5 kg/hr, in a static mixer. The concentration of material I in this homogeneous end product is 1.2%. Panels compression-molded from this product to act as test specimens show no streaks.

#### EXAMPLE 2

##### *Mixing of a paraffin with polystyrene*

In a dynamic mixer, 0.94 kg/hr of a paraffin oil having a viscosity of 2 poise (measured at 30°C and a shear rate of 5 sec<sup>-1</sup>) is mixed with a polymer branch stream flowing at a rate of 3.8 kg/hr and having a viscosity of 10<sup>4</sup> poise (measured at 276°C and a shear rate of 0.07 < D < 1.0

sec<sup>-1</sup>). The resulting premix contains 19.7% of low-viscosity material, which is thus below the solubility limit of paraffin in polystyrene, which is about 30%. The viscosity of this premix is about 1 tenth power less than the viscosity of the main polymer stream. The said main stream, flowing at a rate of 11.7 kg/hr, is mixed with the premix in a static mixer at an average temperature of 280°C. The final concentration of the paraffin in the polystyrene is 5.7%, its distribution therein being homogeneous. The mixing efficiency is again demonstrated by examining compression-molded panels, which show no streaks.

#### WHAT WE CLAIM IS:—

1. A process for mixing liquid materials with each other, which materials show a viscosity difference of at least 2 powers of ten (in terms of poise), by producing a premix in a branch stream of the high viscosity material wherein at least a portion of the low-viscosity material is premixed with a portion of the high-viscosity material in the branch stream at a shear rate of at least 0.1 sec<sup>-1</sup> to form a premix having a content of the low viscosity material of not more than 60% by weight of the premix; the premix is united with the main stream formed by the remainder of the high-viscosity material and the total stream thus formed is divided into partial streams which are spatially rearranged and are intimately mixed at a shear rate of not more than 10<sup>4</sup> sec<sup>-1</sup>.
2. A process as claimed in claim 1, wherein a second high-viscosity material is added via a side stream to the premixing stage.
3. A process as claimed in claim 2, wherein the high-viscosity material provided as a side stream is added to the low-viscosity material alone.
4. A process as claimed in any of claims 1 to 3, wherein a portion of the low-viscosity material is fed directly to the high-viscosity main stream, whilst the remainder of the low-viscosity material is added to the branch stream of high-viscosity material and/or the side stream formed by the second high-viscosity material.
5. A process as claimed in any of claims 1 to 4, wherein a portion of the low-viscosity material is added to the high viscosity material before or during manufacture thereof, for example during polymerization in the case of polymers, the remainder of the low-viscosity material being mixed with the high-viscosity material in the branch stream to form the premix.
6. A process as claimed in any of claims 1 to 5, wherein high-viscosity liquid additives with or without solids contents are added to the branch stream and/or side stream and/or main stream.
7. A process as claimed in any of claims 130

1 to 6 wherein the shear rate in the pre-mixing stage is from  $10^3$  to  $10^4$   $\text{sec}^{-1}$  and the shear rate in the final mixing stage is from  $10^{-2}$  to  $10^2$   $\text{sec}^{-1}$ .

5 8. A process as claimed in any of claims 1 to 7 wherein the higher viscosity material is a polymer.

9. A process for mixing low-viscosity liquid material with high-viscosity liquid material, the viscosity difference being at least two powers of ten (in terms of poise), which comprises mixing a portion of the high-viscosity liquid material with low-viscosity liquid material in a dynamic mixer to form a premix having a viscosity less than two powers of ten (in terms of poise) below the viscosity of the high-viscosity liquid material and mixing the premix with the remainder of the high-viscosity liquid material in a static mixer.

10. A process as claimed in claim 9 in which all the low-viscosity material is fed to the premixing stage.

11. A process for mixing liquid materials substantially as described with reference to any of the foregoing Examples or as illustrated in the accompanying drawing.

12. Apparatus for mixing high-viscosity and low-viscosity liquid materials with each other, comprising a main line for feeding

high-viscosity material, a branch line extending from the main line and equipped with a metering unit for withdrawing a portion of high-viscosity material from the main line, a line for low viscosity material also equipped with a metering unit and joining with the branch line at a junction point downstream of the metering units for feeding low-viscosity material, a dynamic mixer in the branch line downstream of the junction point, a return line for returning premixed material from the dynamic mixer to the main line downstream of the point of connection of the branch line and a static mixer in the main line downstream of the point of connection of the return line.

13. Apparatus as claimed in claim 12 substantially as described in the accompanying drawing.

14. Mixtures of high-viscosity and low-viscosity liquid materials when obtained by a process as claimed in any of claims 1 to 11.

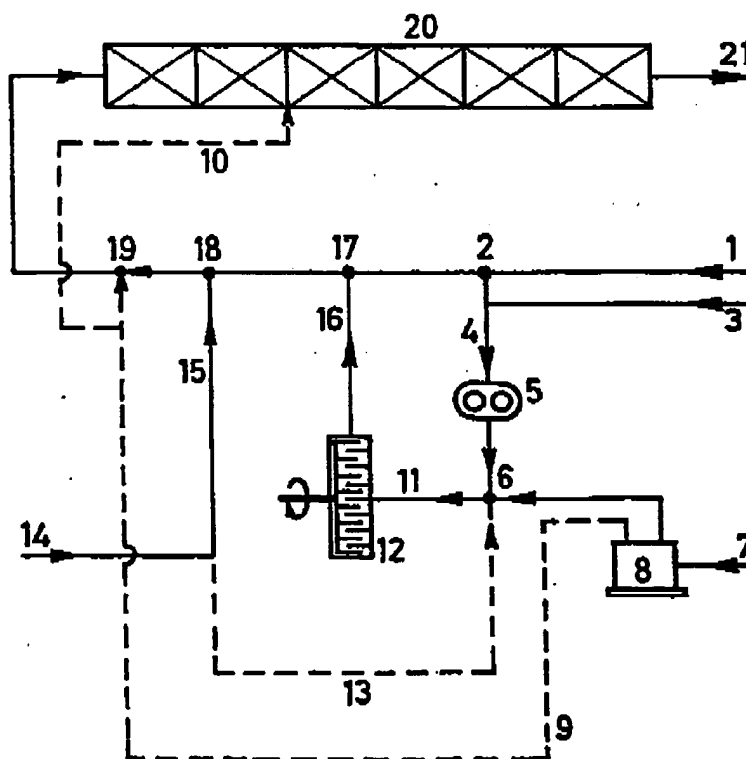
J. Y. & G. W. JOHNSON,

Furnival House,  
14-18 High Holborn,  
London WC1V 6DE.  
Chartered Patent Agents,  
Agents for the Applicants.

Printed for Her Majesty's Stationery Office by The Tweeddale Press Ltd., Berwick-upon-Tweed, 1976.  
Published at the Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.

## COMPLETE SPECIFICATION

This drawing is a reproduction of the Original on a reduced scale.



**This Page is Inserted by IFW Indexing and Scanning  
Operations and is not part of the Official Record**

**BEST AVAILABLE IMAGES**

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

- ☐ **BLACK BORDERS**
- ☐ **IMAGE CUT OFF AT TOP, BOTTOM OR SIDES**
- ☐ **FADED TEXT OR DRAWING**
- ☒ **BLURRED OR ILLEGIBLE TEXT OR DRAWING**
- ☒ **SKEWED/SLANTED IMAGES**
- ☐ **COLOR OR BLACK AND WHITE PHOTOGRAPHS**
- ☐ **GRAY SCALE DOCUMENTS**
- ☐ **LINES OR MARKS ON ORIGINAL DOCUMENT**
- ☒ **REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY**
- ☐ **OTHER:** \_\_\_\_\_

**IMAGES ARE BEST AVAILABLE COPY.**

**As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.**